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Hydrogen sulphide removal in waste water treatment plant by compact oxidative scrubbing in Aquilair PlusTM process

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Abstract Recently, the development of a high voidage contactor, named Aquilair PlusTM, has demonstrated high efficiency for chemical scrubbing of hydrogen sulphide. Liquid and gas phases flow at co-current and high velocity, leading to a great dispersion of the liquid and then to an enhancement of the mass transfer rate by comparison with classical packed towers. This study focused on the results which obtained at semi-industrial scale with the Aquilair PlusTM process on a waste water treatment plant located in France. The scrubbing liquid consisted of a sodium hypochlorite alkaline solution. At once, pressure drop, H₂S removal and reagents consumption were followed. The influence of the superficial gas velocity, liquid-to-gas mass ratio (L/G), pH, hypochlorite concentration of the scrubbing liquid and H₂S inlet concentration was characterised. H₂S removal percentages higher than 90% could be easily achieved with a moderate pressure drop (< 40 mbar). Both hydrodynamic and chemical conditions proved to influence performances. Reagents consumptions slightly higher than the predicted ones were measured.

Keywords Absorption; Chemical scrubbing; Hydrogen sulphide; Odour; Process intensification

INTRODUCTION

Environmental concern pertaining to olfactory nuisances is on the increase. Indeed, odorous pollution is the second reason for complaints after noise. Waste water treatment plants (WWTPs), among other facilities, can cause odour emissions likely to generate significant annoyance in the neighbourhood (Frechen, 2004). Hydrogen sulphide is the predominant odorous molecule associated with sewage (Gostelow et al., 2001) and its formation in sewers has been extensively studied (Matos and de Sousa, 1992; Nielsen et al., 1998). In order to treat these odorous atmospheres, some classical processes are implemented: biofilters can be an alternative but the most common used are packed towers, in which pollutants are removed by chemical scrubbing (Bonnin, 1991; Chen et al., 2001). This technology has been showing good results for many years, but has also revealed high investment and working costs (Couvert et al., 2008b). Indeed, the long residence time necessary for a good functioning of the process and the hydrodynamic behaviour inside the packed column imply the construction of high and wide towers. Comparing to physical absorption, chemical scrubbing offers several advantages. The irreversible degradation of the pollutant in the liquid allows to regenerate the scrubbing solution and to maintain a driving force to achieve mass transfer. Moreover, if the degradation kinetics is fast or instantaneous, a mass transfer acceleration can occur (Coulson and Richardson, 1996; Perry and Green, 1997; Levenspiel, 1999) and the pollutant bulk concentration is equal to 0. This drives to an enhancement of the mass transfer rate. According to the Whitman double film theory, the pollutant is fully consumed in the liquid film at the gas-liquid interface and the H₂S transferred flow (dN in mol.s⁻¹) is given by Eq. (1):

$$dN = k_L a^0 E \frac{P_{H_2S}}{H_{H_2S}} dV \quad (1)$$

Where k_L is the liquid film mass transfer coefficient (m.s^{-1}), a^0 is the volumetric interfacial area ($\text{m}^2.\text{m}^{-3}$), $p_{\text{H}_2\text{S}}$ is the H_2S partial pressure (Pa), $H_{\text{H}_2\text{S}}$ the H_2S Henry's constant and dV an elementary volume. E represents the enhancement factor and is equal to the rate of H_2S transferred when the reaction occurs divided by the rate of H_2S transferred when no reaction occurs. E depends on the Hatta number (Ha), which represents the maximum pollutant conversion in the liquid film compared with its maximum transport through the film (Van Krevelen and Hoftijzer, 1948) and can be estimated by Eq. (2):

$$Ha = \frac{\sqrt{\gamma k C_R D_{\text{H}_2\text{S,L}}}}{k_L} \quad (2)$$

Where γ represents the stoichiometric coefficient of the reaction between the reagents R and H_2S , k is the kinetic constant of the reaction ($\text{M}^{-1}.\text{s}^{-1}$) and $D_{\text{H}_2\text{S,L}}$ is the diffusion coefficient of H_2S in water ($\text{m}^2.\text{s}^{-1}$). Ha higher than 3 indicates that the reaction is fast and occurs in the liquid film where the pollutant is fully consumed. Considering the very short contact time in the Aquilair PlusTM, a high value of E and consequently of Ha , is required.

Recently, a study has emerged to the development of a high voidage contactor, named Aquilair PlusTM, specially adapted to intense chemical scrubbing. The contactor is composed of a patented wire mesh packing structure where liquid and gas flow at co-current and high velocity ($> 10 \text{ m.s}^{-1}$) (Sanchez et al., 2007c). The device allows a great dispersion of the liquid into fine droplets by impact of this phase on the wire mesh, and then to an enhancement of the mass transfer rate by comparison with classical packed towers. At the laboratory scale, it has been shown that an increase of the superficial gas velocity (U_{SG}) and in a minor extent of the superficial liquid velocity (U_{SL}) leads to better interfacial area (a^0) and liquid mass transfer coefficient ($k_L a^0$) due to a higher energy loss (Sanchez et al., 2007a). The gas residence time can be reduced to less than 50 ms instead of about 1 or 2 seconds in packed towers to treat efficiently H_2S in NaOCl caustic aqueous solution (Sanchez et al., 2007b; Couvert et al., 2008a). An economical evaluation demonstrated a reduction of investment and working costs comparing to packed towers comprised between 15 and 40% (Couvert et al., 2008b). These good performances were attributed to fast kinetic of both dissociation reactions due to HO^- and oxidation due to NaOCl . Indeed, H_2S is an acid compound ($\text{p}K_{a1} = 7$ and $\text{p}K_{a2} \approx 12$ at 298 K) (Cadena and Peter, 1988). In NaOCl caustic solution, H_2S reacts with NaOCl to form SO_4^{2-} as a final product. The first step of this degradation could be the dissociation of H_2S in HS^- or its oxidation by hypochlorite. The complete oxidation of one mole of H_2S consumes 4 moles of NaOCl and 2 moles of NaOH (Eq. 3):



A first scale-up of the process was realised with a half-sized industrial pilot plant designed to treat a polluted gas flowrate of $4000 \text{ m}^3.\text{h}^{-1}$ (Couvert et al., 2008b). Nevertheless, results were quite disappointing by comparison to the laboratory, since lower H_2S removals were obtained for similar pressure drops. This phenomenon could be explained by lower wall effects in the industrial plant, which implied a higher U_{SG} to achieve a fully dispersed flow. Consequently, several improvements were realised, the main ones were the decrease of the contactor diameter from 300 mm to 200 mm and the design of a new wire mesh packing structure. The decrease of the diameter was done to reach higher U_{SG} without changing the fan. The new packing structure presents a high void fraction ($> 99.9\%$) but the spatial arrangement of the mesh offers a higher tortuosity.

This study focuses on the results achieved after these changes. At once, the pressure drop (ΔP), the H_2S removal and the reagent consumption were determined. The influence of the superficial gas velocity (U_{SG}), liquid-to-gas mass ratio (L/G), pH , sodium hypochlorite concentration of the scrubbing liquid ($[\text{NaOCl}]$) and H_2S inlet concentration ($[\text{H}_2\text{S}]_{\text{G,i}}$) was characterised.

MATERIAL AND METHODS

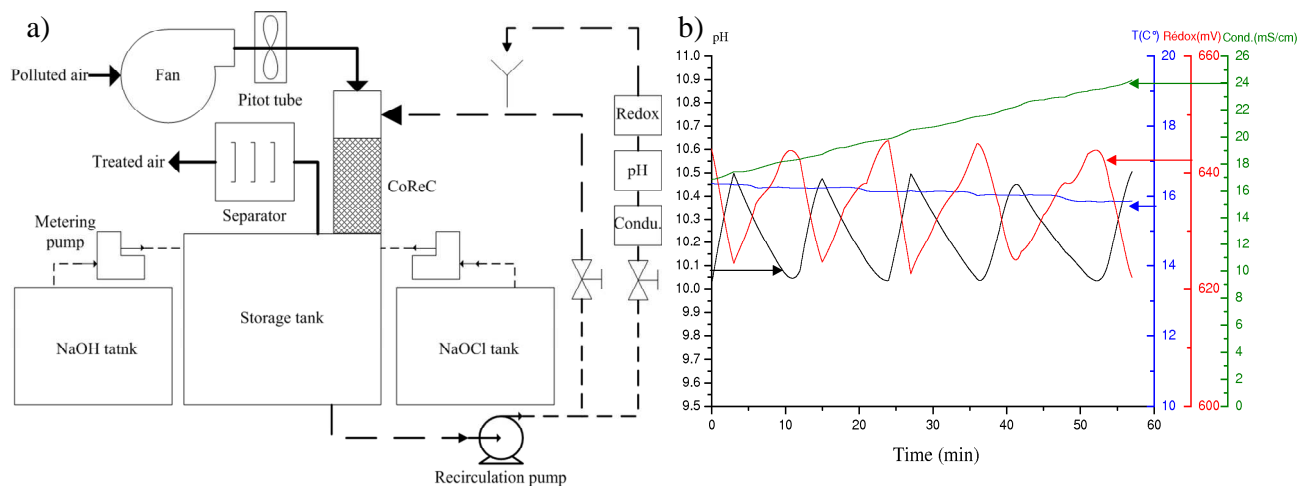
Contactor

The contactor is the key element of the process. It consists of a wire mesh structure of 200 mm diameter (void fraction > 99.9%). The initial height of the packing was 600 mm but after the first experiments, the packing was compressed on 500 mm.

Reactor Set-up (Figure 1a)

The pilot was controlled thanks to an Allen Bradley automation system (Rockwell automation, USA) and piloted with a PcVue supervision. The polluted air was pumped from the extraction pipe of a French WWTP with a fan (AEIB, France). By opening a valve placed before the fan, it was possible to dilute polluted air with clean air to study influence of the inlet H_2S concentration ($[H_2S]_{G,i}$ in ppmv: Part Per Million Volumic). The gas flowrate Q_G was measured and controlled thanks to a Pitot Tube Deltabar S PMD70 (Endress Hauser, Germany) and a frequency variator (Allen Bradley). The liquid injection upstream the contactor was insured by a spray nozzle SpiralJet (Spraying System, USA). Pressure upstream and downstream the contactor was measured thanks to two pressure sensors Cerabar S PMC71 (Endress Hauser, Germany). A 400 L tank allowing to store the scrubbing liquid and to separate the gas-liquid mixture was placed downstream the contactor. A droplet separator has been added downstream the tank before emission in atmosphere (Horus Environnement, France). The storage tank was filled with ground water previously softened. Reagents (NaOCl and NaOH provided by Brenntag, Germany) were pumped from 1000 L tanks to the storage tank thanks to two Gamma L metering pumps (Prominent, USA). The scrubbing liquid was recirculated to the top of the contactor through a MDFL pump (Iwaki, Japan) and the liquid flowrate was controlled thanks to an electromagnetic flowmeter Promag 10P (Endress Hauser, Germany). Moreover, the storage tank was equipped with two level sensors BSM (Bamo, France).

Figure 1. a) Reactor set-up b) Regulated parameters during a typical experiment.



Regulation

A bypass located after the recirculation pump allows the measurement of the pH, redox, temperature and conductivity of the scrubbing liquid (pH and temperature sensor: Orbisint CPS 11; redox sensor: Orbisint CPS 12; conductivity sensor: Indumax CLS 50; all provided by Endress Hauser). The pH and NaOCl concentration of the scrubbing liquid, clean water supply and drain were regulated respectively by (Figure 1b):

- pH measurement (when the low threshold is reached, injection of NaOH begins until the high threshold is reached);
- Redox measurement (when the low threshold is reached, injection of NaOCl begins until the high threshold is reached);

-Conductivity measurement (when the high threshold is reached, drain of the storage tank begins until the low level is reached. Then, clean water is supplied until the high level is reached).

Measurement methods

The concentration of the NaOH commercial solution was regularly checked by colorimetric titration (phenolphthalein) with sulphuric acid and the concentration of the commercial solution of NaOCl by the iodometric method. Moreover, at the beginning and the end of each experiment, the concentration of the scrubbing liquid in the storage tank was measured. The H₂S concentration was continuously measured upstream and downstream the Aquilair PlusTM by two electrochemical sensors Polytron (Dräger, Germany).

RESULTS AND DISCUSSION

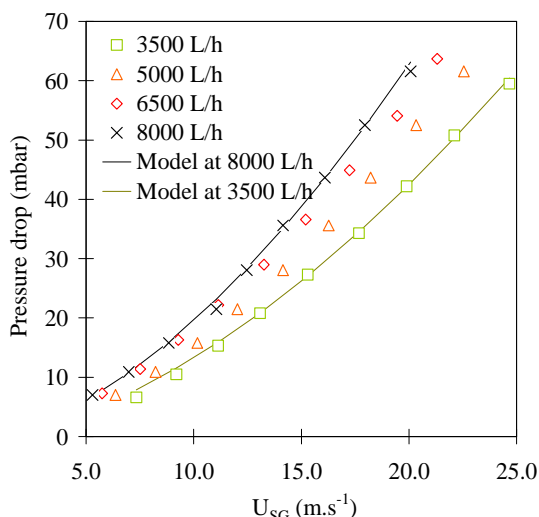
Pressure drop inside the contactor

The Pressure drop across the contactor was measured for U_{SG} varying between 5.3 and 24.7 m.s⁻¹ (i.e. for a gas flowrate Q_G between 600 and 2800 m³.h⁻¹) and four liquid flowrates Q_L (Fig. 2). This parameter strongly increased with U_{SG} and with a minor extent with Q_L. As gas is the continuous phase, it plays a major role. Consequently, small variations of U_{SG} can generate important variations of the pressure drop, which is an important criterion to consider for the process economic viability. The pressure drop per reactor height unit (ΔP/H) could be well correlated to the gas and liquid superficial velocities U_{SG} and U_{SL} (Sanchez et al., 2007a):

$$\frac{\Delta P}{H} (\text{mbar.m}^{-1}) = 2.857 \times U_{SG}^{1.68} \times (U_{SL})^{0.47} = 0.119 \times U_{SG}^{2.15} \times \left(\frac{L}{G}\right)^{0.47} \quad (4)$$

The correlation coefficient between the experimental and the predicted pressure drops is 99.94 %. The exponent on U_{SG} of 1.68 confirms high dependence between the pressure drop and U_{SG}.

Figure 2. Pressure drop inside Aquilair PlusTM versus U_{SG} for different Q_L.



Through the transparent PVC pipe reactor, a strong dispersion of the liquid into small droplets was observed for U_{SG} higher than 13-14 m.s⁻¹. It is interesting to notice that when the liquid flowrate increased, this dispersion regime appeared for a lower U_{SG}.

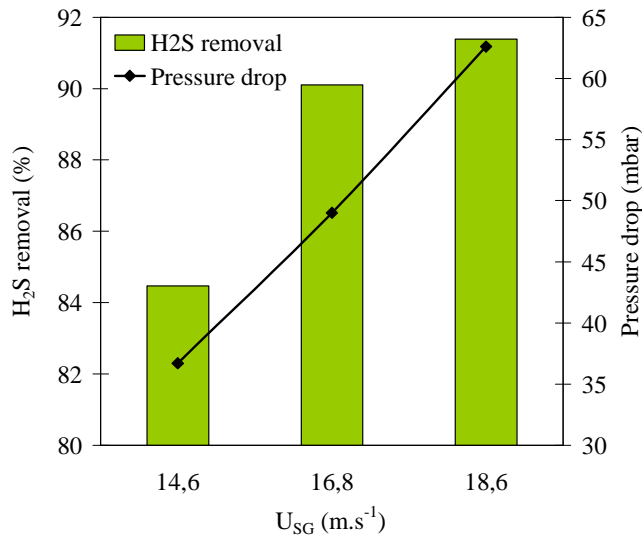
H₂S removal

Choice of the working pH range. Experiments carried out for pH varying between 9.5 and 11 showed that the H₂S removal is not significantly different (not presented here). However, for a pH

close to 9.5, the formation of a yellow precipitate of colloidal sulphur was observed whereas for a pH close to 11, the formation of a white precipitate of carbonates could be noticed (due to the reaction between NaOH and CO₂ present in atmospheric air). Consequently, all the following experiments were performed with a pH ranging between 10 and 10.5.

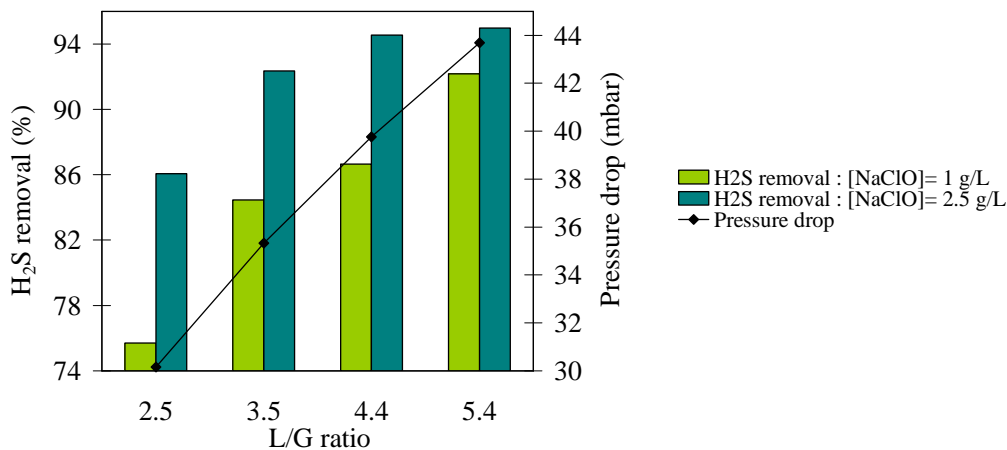
Influence of the superficial gas velocity on the H₂S removal. Figure 3 shows that a higher U_{SG} induces a better H₂S removal. When U_{SG} increases, it induces a higher dispersion of the liquid and a higher mixing of both phases. As a consequence, droplets are smaller, which means that a⁰ increases, and k_L is higher. For example, at the laboratory, it has been shown that a⁰ and k_L were respectively proportional to U_{SG}^{0.72} and U_{SG}^{1.11} (Sanchez et al., 2007a). According to Eq. (1), the H₂S transferred flow is proportional to k_L and a⁰, which explain the increase of the H₂S removal with U_{SG}. Nevertheless, working at high superficial gas velocities is not a cost effective solution as the pressure drop climbs. Consequently, a superficial gas velocity ranging between 14 and 15 m.s⁻¹ (Q_G from 1600 to 1700 m³.h⁻¹) was selected for all the next experiments.

Figure 3. H₂S removal and pressure drop versus U_{SG} (L/G = 3.5; 40 < [H₂S]_{G,i} < 50 ppmv; [NaOCl] ≈ 1.5 g.L⁻¹; 10 < pH < 10.5).



Influence of the L/G ratio on the H₂S removal.

Figure 4. H₂S removal and pressure drop versus L/G ratio (U_{SG} = 15 m.s⁻¹; 40 < [H₂S]_{G,i} < 50 ppmv; 10 < pH < 10.5).

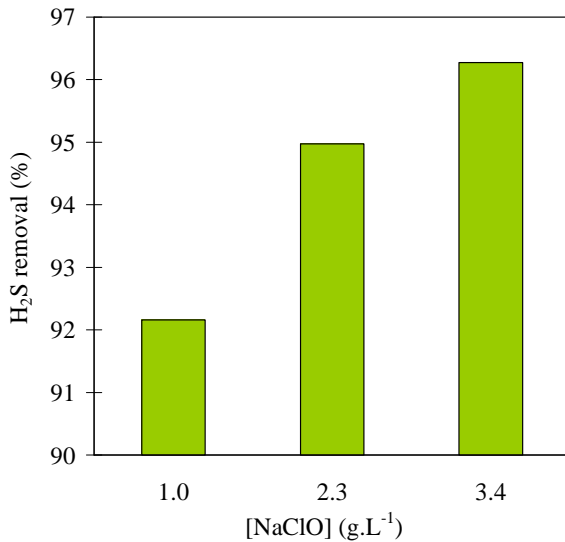


To achieve the H₂S chemical scrubbing in a packed column, L/G is generally close to 3, which

means that the volumetric gas flowrate is approximately 300 times greater than the volumetric liquid flowrate. Figure 4 demonstrates that an increase of L/G at a given gas flowrate induces a significant better H₂S removal. This evolution could be attributed to better hydrodynamic and thermodynamic conditions. Sanchez et al. (2007a) demonstrated at the laboratory scale that k_{La}^0 increases with the liquid velocity (or flowrate). Moreover, a higher L/G implies that more H₂S can be solubilised in the liquid phase and that more reagents are present in the contactor. However, increasing the liquid flowrate generates a higher pressure drop and consumes more electrical power for the recirculation, which leads to a more expensive operation. Experiments done at moderate U_{SG} (14.1 m.s⁻¹) and with L/G = 5.4 allowed to reach a H₂S removal close to 95 % with a rather low pressure drop of 39 mbar (not represented here).

Influence of the sodium hypochlorite concentration on the H₂S removal. As well as hydrodynamic conditions, chemical conditions can notably influence the H₂S removal. No significant variation of the H₂S removal was observed when the pH of the scrubbing liquid was low (pH = 10) or high (pH = 11). Nevertheless, Figure 5 demonstrates a positive influence of the sodium hypochlorite concentration on the H₂S removal. This result is particularly interesting considering that it is a low cost operating solution.

Figure 5. H₂S removal versus [NaOCl] ($U_{SG} = 15 \text{ m.s}^{-1}$; L/G = 5.4; $40 < [H_2S]_{G,i} < 50 \text{ ppmv}$; $10 < \text{pH} < 10.5$; $\Delta P = 44 \text{ mbar}$).



Generally, during chemical scrubbing of acidic compounds, the dissociation reactions carried out are more efficient than oxidation reactions for enhancing mass transfer due to faster kinetics (Chen et al., 2001; Couvert et al., 2006). In this case, the role of the oxidant is to degrade the pollutant in order to recycle the scrubbing liquid and to maintain a driving force for the pollutant mass transfer. Nevertheless, during the H₂S removal in the presence of sodium hypochlorite, the oxidation reaction could be more efficient than the dissociation reaction to accelerate mass transfer. Indeed, the reaction between H₂S and ClO⁻ at basic pH is really fast according to the kinetic constant of $1.8 \times 10^8 \text{ L.mol}^{-1}.\text{s}^{-1}$ found by Bonnin (1991). This could be easily demonstrated by comparing the Hatta numbers of each reaction (Eq. 5-8):

$$Ha_{ox} = \frac{\sqrt{k_{ox,H_2S} \cdot [ClO^-] \cdot D_{H_2S}}}{k_L}$$

$$(5) \text{ and } Ha_{diss} = \frac{\sqrt{k_{diss,H_2S} \cdot [HO^-] \cdot D_{H_2S}}}{k_L} \quad (6)$$

$$\Rightarrow \left(\frac{Ha_{diss}}{Ha_{ox}} \right)^2 = \frac{k_{diss,H_2S} \cdot [HO^-]}{k_{ox,H_2S} \cdot [ClO^-]} \quad (7)$$

$k_{\text{diss},\text{H}_2\text{S}}$ can be taken equal to $5.4 \times 10^8 \text{ L} \cdot \text{mol}^{-1} \cdot \text{s}^{-1}$ (Bonnin, 1991). In a first approximation, without considering the ionic strength of the scrubbing liquid, $[\text{HO}^-]$ is equal to $10^{\text{pH}-14}$ and varies between 1.0×10^{-4} and $3.2 \times 10^{-4} \text{ mol} \cdot \text{L}^{-1}$. Considering $[\text{ClO}^-] = 1 \text{ gCl} \cdot \text{L}^{-1}$, it leads to:

$$0.01 < \left(\frac{\text{Ha}_{\text{diss}}}{\text{Ha}_{\text{ox}}} \right)^2 < 0.03 \quad (8)$$

When two distinct reactions can occur, the global Ha (Ha_{glo}) can be estimated by Eq. (9):

$$\text{Ha}_{\text{glo}}^2 = \text{Ha}_{\text{ox}}^2 + \text{Ha}_{\text{diss}}^2 = \text{Ha}_{\text{ox}}^2 \left(1 + \left(\frac{\text{Ha}_{\text{diss}}}{\text{Ha}_{\text{ox}}} \right)^2 \right) \approx \text{Ha}_{\text{ox}}^2 \quad (9)$$

Consequently, the mass transfer enhancement in our chemical conditions can be attributed to oxidation reaction. Nevertheless, HO^- anions play a fundamental role during the scrubbing step thanks to reaction (3). At the laboratory scale, k_L was close to $1 \times 10^{-4} \text{ m} \cdot \text{s}^{-1}$. $D_{\text{H}_2\text{S},\text{L}}$, estimated thanks to the Wilke and Chang correlation (Perry and Green, 1997), is equal to $1.41 \times 10^{-9} \text{ m}^2 \cdot \text{s}^{-1}$. This leads to a Hatta number of approximately 850 for $[\text{NaOCl}] = 1 \text{ gCl} \cdot \text{L}^{-1}$. This confirms that H_2S reacts completely in the liquid film, and that its removal strongly depends on the NaOCl concentration.

Influence of the H_2S gaseous concentration. Several experiments were done for $U_{\text{SG}} = 14.1 \text{ m} \cdot \text{s}^{-1}$, $L/G = 5.5$, $10 < \text{pH} < 10.5$ and $[\text{NaOCl}] \approx 3 \text{ g} \cdot \text{L}^{-1}$, but with $[\text{H}_2\text{S}]_{\text{G},i}$ varying from 6 to 80 ppmv. The H_2S removal was not significantly different and close to 96%. It was not possible to study a lower $[\text{H}_2\text{S}]_{\text{G},i}$ because the detection level of the electrochemical sensor Polytron was reached at the outlet. With a classical H_2S inlet concentration in WWTP of 5-6 ppmv, it is consequently possible to reach an H_2S outlet concentration ranging between 0.1 and 0.2 ppmv, which is acceptable.

Reagent consumption of the process

The NaOCl and NaOH consumptions were estimated considering the functioning time of each reagent metering pump, the concentration of each reagent solution and the duration of each experiment (1 hour on average). The consumption was about 6.1 mol of NaOCl and 3.0 mol of NaOH per mol of H_2S transferred whereas the theoretical consumptions are equal to 4 and 2 moles respectively. This over-consumption could not be justified by the absorption/reaction of other species since H_2S was the only one detected. Indeed, a sample of the inlet polluted gas was analysed, after concentration on Tenax Tube, by Gas chromatography (Agilent 300) coupled with Mass Detection (Agilent 5973N) (Injector temperature: 90°C ; column temp.: 110°C for 30 psi; carrier gas: helium; mass detection in scan mode; source temp.: 250°C ; column OV1: $40\text{m} \times 0.15\text{mm} \times 2\mu\text{m}$). At the laboratory scale, the reagent consumption was lower than the theoretical one (Couvert et al., 2008a). Nevertheless, no recirculation of the scrubbing liquid was implemented and the consumption was calculated between inlet and outlet of the contactor. As the residence time is identical between the contactors of both studies (50 ms), it seems to be obvious that the over-consumption observed in the pilot plant takes place in the storage tank. Indeed, in this part of the pilot ($\varnothing = 900 \text{ mm}$), which is dedicated to the gas-liquid separation, the interface between gas and liquid is high, and mass transfer between phase can occur. Then, the NaOH consumption could be attributed to the absorption of CO_2 present in atmospheric air above the scrubbing liquid in the tank and the NaOCl consumption to stripping from the liquid to the air. Some improvements of the storage tank design should allow to decrease the over consumption in limiting the interface between the treated gas and the scrubbing liquid.

CONCLUSION

A new compact chemical scrubber was implemented in a WWTPs. Results showed that both hydrodynamic and chemical conditions significantly influence the H_2S removal. An interesting economic compromise was deduced for a moderate gas velocity and a high hypochlorite

concentration. A great advantage of this contactor is that high variations of the gas flowrate can be supported without any risk of flooding. This study demonstrated the feasibility of compact scrubbing at large scale where there is lower wall effect than at the laboratory scale. The new packing has not been optimised yet and some improvement of the structure would certainly offer a lower pressure drop for a similar mass transfer rate.

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